LABORATORY Spotlight
The National High Magnetic Field Laboratory
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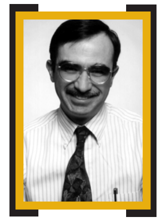
NHMFL NMR Spectroscopy and Imaging Update



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This program has a mission to develop technology and applications at the highest magnetic fields through both in-house and external user activities. This is a very broad mission in solution and solid-state NMR spectroscopy as well as imaging and diffusion measurements. Unique capabilities are being developed in all of these arenas using superconducting magnets up to 19.6 T and resistive magnets up to 25 T or 1.066 GHz. Condensed matter NMR has extended this range up to 45 T in the hybrid magnet at the NHMFL. These magnets vary from very narrow bore (31 mm) to whole body dimensions, and they are located at two sites—the NHMFL in Tallahassee and at the UF McKnight Brain Institute in Gainesville. Here, we highlight two new capabilities made possible by the efforts of Ago Samosan from the National Institute of Chemical and Biophysics in Estonia and Andrei Samoilenko from the Chemical and Physics Institute, Russian Academy of Sciences in collaboration with Zhehong Gan, Bill Brey, Riqiang Fu, and Peter Gor'kov at the NHMFL. Samosan and Samoilenko are supported by the Newly Independent States Visitor's Program, a supplement to the NSF core grant to the NHMFL.

Odd-halves quadrupole nuclei represent great challenges and opportunities. To spectrally resolve chemically unique sites, many approaches have been developed to defeat second order broadening of the central transition due to the similar magnitude of the quadrupolar and Zeeman interactions. Higher fields directly reduce this broadening effect, as shown by many studies including recent experiments up to 25 T as described in the Fall 2000 *NHMFL Reports*. In this work, Zhehong Gan collaborated with Dominique Massiot from CNRS in Orleans, France to study aluminum sites in $^{9}Al_2O_3$ + $^{2}B_2O_3$. Four aluminum sites are much better resolved at 25 T than even at 19.6 T in magic angle spinning spectra. There are additional ways to enhance these quadrupolar spectra. One very successful approach for nuclei with small or modest quadrupole couplings involves multiple quantum magic angle spinning (MQMAS). A recently developed and high sensitivity approach correlates the satellite transitions in two-dimensional MAS spectra (STMAS).[1] Now, an approach originally developed by Samosan, Lippmaa, and Pines[2] involving rotation about two octahedral axes simultaneously has been implemented at the NHMFL through Ago Samosan. Such a technology operating in high field magnets has the potential to achieve very high resolution spectra even of nuclei with very large quadrupole interactions. The ^{17}O DOR (Double Rotation Spectroscopy) spectrum of KTP in Fig. 1 demonstrates the high spectral resolution from the DOR and the high field that resolves eight Ti-O-P sites in a chemical shift range less than 10 ppm.

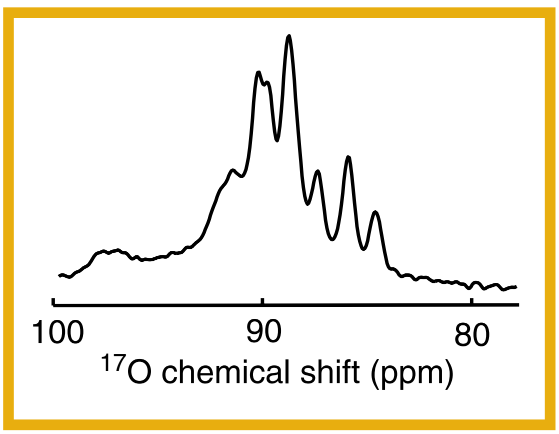

Figure 1. DOR (1400 Hz) spectrum of ^{17}O-enriched KTP at 16.9 T. Sample courtesy from Ray Dupree, University of Warwick/UK.

Imaging with NMR in the solid state suffers from a variety of problems, but one of the biggest problems is that linewidths are very broad. Efforts to image solids using MAS and synchronized acquisition has been demonstrated in several laboratories. Another approach to this problem is to use much larger field gradients than is normally used. A way to achieve this is to take advantage of the stray field of an NMR magnet.[3] In a 19.6 T magnet approximately 20 cm below the center of the field is a gradient of 75 T/m at a field strength of 11.7 T. This is equivalent to more than 3 kHz in ^{1}H frequency per micron giving rise to the potential for excellent spatial resolution even though resonances may be very broad. While it is not possible to pulse such gradients, it is possible to move the sample with respect to the gradient. In Fig. 2, we show one dimensional resolution of approximately 20 μm. Because the very large field gradients can dominate substantial magnetic susceptibility effects, it is possible to image even conducting samples.

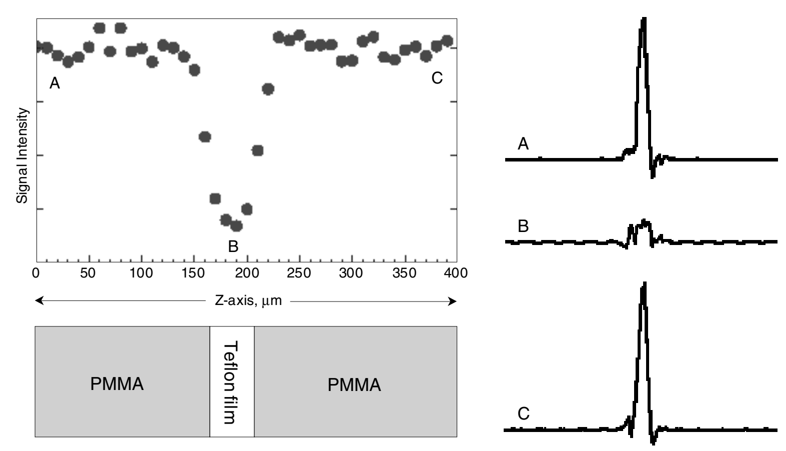

Figure 2. *(left)* 1D proton image of PMMA phantom with 39 µm Teflon film; *(right)* ^{1}H spectra from points A, B, and C in the image.

The NHMFL is pleased to make these unique capabilities available to both national and international user communities. The DOR capability is available at a variety of superconducting fields up to 16.9 T and in resistive magnets up to 25 T. STRAFI is available today at 19.6 T, but the program has interests in adapting this technology to the resistive magnets. Those who are interested in using DOR should contact Zhehong Gan (*gan@magnet.fsu.edu*) and those interested in using STRAFI should contact Riqiang Fu (*rfu@magnet.fsu.edu*). For information on a wide range of other capabilities, please visit our Web site at *http://nmr.magnet.fsu.edu/*.

1 Gan, Z., *J. Am. Chem. Soc.*, **122**, 3243, (2000).
2 Samosan, *et al.*, *Mol. Phys.*, **65**, 1013, (1988).
3 Samoilenko, *et al.*, *JETP Lett*, (1988).